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(54) Title: LAMINATE WAFERS

(57) Abstract

A laminate optical article including a first outer layer; a second outer layer; and an inner layer positioned between said first and second outer layers, the inner layer providing a desirable optical property to the laminate optical article.

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LAMINATE WAFERS

The present invention relates to the manufacture of plastic optical articles such as sunglass lenses, ophthalmic lenses and the like. In particular, the present invention relates to the manufacture of laminate optical articles.

It is known in the prior art that in the manufacture of optical articles it is often desirable to provide additional properties to the articles, for example polarisation, UV inhibition, photochromism, electrochromism, or colouration of lenses.

Such properties are normally provided by the addition of a coating or surface treatment to the finished lens. However, such treatments are difficult and expensive to apply, and may be removed from the optical surface by scratching, wearing or the like.

Further, for example, it is known in the prior art to incorporate photochromic dyes into a plastic lens via surface imbibation techniques. See for example United States Patent 5,130,353, or United States Patent 5,185,390, which describe the inclusion of photochromic dyes into the subsurface region of a plastic lens by first positioning a polymeric or other carrier or photochromic dye physically against the surface of the lens and then using heat to cause the dyes to diffuse into the lens. Such techniques are both difficult and expensive, and require careful control of the depth of absorption in order to achieve a constant colour for the lens.

Further, it has been proposed in the prior art, e.g. in Japanese Patent 1259591 (1984) and Japanese Patent application 5181016 (1993), to introduce a film having desirable properties by placing a foil into a mould and cast polymerising a lens. Numerous problems have been associated with this art, however, including the formation of bubbles, haze and visible stress birefringence in the finished lens.

It would therefore be a significant advance in the art to provide a process for improving optical properties which was easy to handle and produced a product of enhanced durability.

It is accordingly an object of the present invention to overcome, or at least alleviate, one or more of the difficulties or deficiencies related to the prior art.

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Accordingly, in a first aspect of the present invention there is provided a laminate optical article including

a first outer layer;

a second outer layer; and

an inner layer positioned between said first and second outer layers, the inner layer providing a desirable optical property to the laminate optical article.

The laminate optical article may be characterised by improved durability and ease of handling. The laminate structure provides a product the components of which are of relatively simple geometry and are thus easy to manufacture.

The first outer layer may form a front or back wafer of a laminate optical lens. The first outer layer may include an optical surface and an interface or mating surface. The optical surface may have different optical powers along different meridians. Alternatively the optical surface may include an aspheric bifocal, tri-focal or progressive multi-focal region or regions.

The first outer layer may be formed from any suitable material. A mineral glass or optical polymeric material may be used. The polymeric material may be of any suitable type. The polymeric material may include a thermoplastic or thermoset material. A material of the polycarbonate type, may be used. The laminate optical article is particularly suitable in polycarbonate applications. The laminate optical article may reduce or eliminate difficulties associated with the high stress co-efficient of birefringence associated with polycarbonates. The consequence of this is that when polycarbonate is moulded utilising conventional processes, flow lines and areas of stress are highly visible, for example when viewed through a polarising film.

Alternatively, the polymeric material may be of the diallyl glycol carbonate type, for example the product sold under the trade designation CR-39 (CR-39 is a Registered Trade Mark of PPG Industries, Inc.).

The polymeric material may be formed from cross-linkable polymeric casting compositions, for example as described in applicants United States Patent 4,912,155, United States Patent Application No. 07/781,392, Australian Patent Applications 50581/93 and 50582/93, and European Patent Specification 453159A2, the entire disclosures of which are incorporated herein by reference.

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Such cross-linkable polymeric casting compositions may include a diacrylate or dimethacrylate monomer (such as polyoxyalkylene glycol diacrylate or dimethacrylate or a bisphenol fluorene diacrylate or dimethacrylate) and a polymerisable comonomer, e.g. methacrylates, acrylates, vinyls, vinyl ethers, allyls, aromatic olefins, ethers, polythiols and the like.

For example, in Australian Patent Application 81216/87, the entire disclosure of which is incorporated herein by reference, applicant describes a cross-linkable coating composition including at least polyoxyalkylene glycol diacrylate or dimethacrylate and at least one poly functional unsaturated cross-linking agent.

Further, in Australian Patent Application 75160/91, the entire disclosure of which is incorporated herein by reference, applicant describes a polyoxyalkylene glycol diacrylate or dimethacrylate; a monomer including a recurring unit derived from at least one radical-polymerisable bisphenol monomer capable of forming a homopolymer having a high refractive index of more than 1.55; and a urethane monomer having 2 to 6 terminal groups selected from a group comprising acrylic and methacrylic groups.

Such polymeric formulations are UV cured or cured by a combination of UV and thermal treatment. The range of optical lenses sold under the trade designations "Spectralite" by Applicants have been found to be suitable.

The polymeric material may include a photochromic dye which may, for example, be added to the monomer formulation used to produce the polymeric material.

The second outer layer of the laminate optical article may be of any suitable type. The second outer layer may form a protective coating for the inner layer described above. The protective coating may include one or more of the groups consisting of an abrasion resistant component, a water resistant component and an antistatic component. A peel-off protective layer may be used. The protective coating may be formed of a polymeric material. A vinyl polymer material may be used. A vinyl chloride/vinyl acetate copolymer is preferred. The protective coating may further include conventional components including plasticisers, anti-static agents and the like.

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The laminate optical article so formed may be packaged for later lamination to, for example, a front or back lens wafer. A water-barrier resistant package may be used. Such packages may control relative humidity such that the stability of curvature of the lens wafers is retained. A package of the type described in United States Patent 5,323,192 to applicants, the entire disclosure of which is incorporated herein by reference, may be used.

Alternatively, the second outer layer may form a complementary back or front wafer of a laminate optical lens. The second optical layer may form a complementary back or front wafer to the first outer layer. The second outer layer may be formed in a complementary though reverse manner to the first outer layer.

Accordingly, in a preferred aspect of the present invention there is provided a laminate optical article including

a front lens wafer formed from a optical polymeric material;

a second complementary back lens wafer formed from a optical polymeric material; and

an inner layer positioned between said front and back wafer and having a shape complementary to a surface complementary to a surface of the front and/or back wafer, the inner layer providing a desirable optical property to the laminate optical article.

If desired, there may be a distribution of distance power and cylinder between the front and back lens wafers. Alternatively, the back lens wafer may be relatively thick, the laminate optical article forming a semi-finished lens.

The inner layer may be of any suitable type. The inner layer may include a polymeric film having desirable optical properties. The inner layer may include a foil, that is a film having structural integrity.

The inner layer may be of any suitable size and shape. The inner layer may be formed to have a shape complementary to a surface of the first outer layer and/or second outer layer. The inner layer may be moulded, cast, blown or otherwise shaped to have a complementary surface to the first outer layer and/or second outer layer.

The inner layer may be trimmed in situ or, more preferably may be pre-cut to size. The edge of the inner layer may include a return flange to aid in

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dimensional stability and handling.

The inner layer may be formed to substantially correspond to the shape of an interface or mating surface of the first outer layer and/or second outer layer. Where the interface or mating surface is a curved surface, the inner layer may have a curvature which substantially corresponds to the curvature of the inner surface of the first and/or second outer layer. A typical curvature required would be of the order of approximately 4 to 8 dioptre.

It will be understood that in this preferred form since the interface surface(s) of the front lens wafer and/or second complementary back lens wafer can be kept relatively simple, the formation of the inner layer is also relatively simple and can be controlled within very close tolerances. This may reduce or prevent formation of bubbles and other optical distortions.

The desirable optical properties may be inherent in the nature of the polymeric film. For example, a vinyl alcohol polymer may be used. A hydrolysed polyvinyl acetate (polyvinyl alcohol) may be used. The polyvinyl alcohol may be highly oriented to produce a polarising film or foil. The polyvinyl alcohol may be iodine impregnated. A polyvinyl acetate or polyvinyl butyrate polymer may be included. Such a polymer may include an active light polarising films. The polyvinyl alcohol film or foil may be provided, in a preferred form, in a cellulose acetate butyrate or like laminate.

Alternatively, the inner layer may include a polymeric film or foil having incorporated therein an optically active component.

The polymeric film or foil may be of any suitable optically transparent or transmissible material. The polymeric film may be selected from optically transparent acrylics, vinyls, allyls, esters and the like.

Where the inner layer exhibits UV absorbing characteristics, for example with a polarising film or foil, it is preferred to select a material which does not exhibit a sharp UV cut off. Such a UV cut off may present difficulties where a UV curable adhesive is used in the lamination process. For example UV cure may be conducted around 365 nm so low transmission in this region may result in poor laminate adhesion.

Alternatively where a sharp UV cut off is required, for example in

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sunglass applications, a visible light curable adhesive may be used.

Similarly, the laminate optical article may be formed from a polymeric material exhibiting a UV cutoff. Such polymeric materials are disclosed in International Patent Application PCT/AU96/00705 "Light Transmissible Article with Reduced Ultraviolet Transmission" to applicants, the entire disclosure of which is incorporated herein by reference.

Where a photochromic film is required, the polymeric film may be formed from cross-linkable polymeric casting compositions, such as a blend of polyoxyalkylene glycol diacrylate or dimethacrylate together with radical-polymerisable bisphenol monomer and a urethane monomer having 2 to 6 terminal groups selected from a group comprising acrylic or methacrylic groups. A preferred composition is a blend of polyoxyalkylene glycol dimethacrylate with urethane methacrylate.

The optically active component may be selected from one or more of the group consisting of light polarising materials, ultra-violet inhibitors, photochromic materials, electrochromic materials and other pigments or dyes.

The pigment(s) or dye(s) including photochromic dye(s) may be selected from one or more of the group consisting of anthraquinones, phthalocyanines, spiro-oxazines, chromenes, pyrans including spiro-pyrans and fulgides. The photochromic dye(s) may be incorporated into the monomer formulation used to produce the polymer of the inner layer.

Examples of preferred photochromic dyes may be selected from one or more of the group consisting of

- 1,3-dihydrospiro[2H-anthra[2,3-d]imidazole-2,1'-cyclohexane]-5,10-dione
- 25 1,3-dihydrospiro[2H-anthra[2,3-d]imidazole-2,1'-cyclohexane]-6,11-dione
 - 1,3-dihydro-4-(phenylthio)spiro[2H-anthra'1,2-diimidazole-2,1'-cyclohexane] 6,11-dione
 - 1,3-dihydrospiro[2-H-anthra[1,2-d]imidazole-2,1'-cycloheptane]-6,11-dione
 - 1,3,3-trimethylspiro'indole-2,3'-[3H]naphtho[2,1-b]-1,4-oxazine]
- 30 2-methyl-3,3'-spiro-bi-[3H-naphtho[2,1-b]pyran] (2-Me)
 - 2-phenyl-3-methyl-7-methoxy-8'-nitrospiro[4H-1-benzopyran-4,3'-[3H]-naphtho][2,1-b]pyran

- Spiro[2H-1-benzopyran-2,9'-xanthene]
- 8-methoxy-1',3'-dimethylspiro(2H-1-benzopyran-2,2'-(1'H)-quinoline
- 2,2'-Spiro-bi-[2H-1-benzopyran]
- 5'-amino-1',3',3'-trimethylspiro[2H-1-benzopyran-2,2'-indoline
- Ethyl-β-methyl-β-(3',3'-dimethyl-6-nitrospiro(2H-1-benzopyran-2,2'-indolin-1'yl)-propenoate
 - (1,3-propanediyl)bis[3',3'-dimethyl-6-nitrospiro[2H-1-benzopyran-2,2'-indoline]
 - 3.3'-dimethyl-6-nitrospiro[2H-1-benzopyrao-2,2'-benzoxazoline]
- 6'-methylthio-3,3'-dimethyl-8-methoxy-6-nitrospiro[2H-1-benzopyran-2,2'-benzothiozoline]
 - (1,2-ethanediyl)bis[8-methoxy-3-methyl-6-nitrospiro[2H-1-benzopyran-2,2'-benzothiozoline]
- N-N'-bis(3,3'-dimethyl-6-nitrospiro[2H-1-benzopyran-2,2'(3'H)-benzothioazol 6'-yl)decanediamide
 - $-\alpha$ -(2,5-dimethyl-3-furyl)thylidene(Z)-ethylidenesuccinic anhydride; α -(2,5-dimethyl-3-furyl)- α ', δ -dimethylfulgide
 - 2,5-diphenyl-4-(2'-chlorophenyl)imidazole
 - [(2',4'-dinitrophenyl)methyl]-1H-benzimidazole
- 20 N-N-diethyl-2-phenyl-2H-phenanthro[9,10-d]imidazol-2-amine
 - 2-Nitro-3-aminofluorene
 - 2-amino-4-(2'-furanyl)-6H-1,3-thiazine-6-thione
 - 3,3-di(4-methoxyphenyl)-6-morpholino-3H-naphtho[2,1-b]pyran (Reversacol Corn Yellow)
- 25 3,3-di(4-methoxyphenyl)-3H-naphtho[2,1-b]pyran (Reversacol Tangerine)
 - 2,2-di(4-methoxyphenyl)-5,6-dimethyl-2H-naphtho[2,1-b]pyran (Reversacol Berry Red)
 - 1,3-dihydro-3,3-dimethyl-1-isobutyl-spiro[2H-indole-2,3'-[3H]naphth[2,1-b][1,4]oxazine] (Reversacol Oxford Blue)
- 1,3-dihydro-3,3-trimethyl-1-neopentyl-6'-(4-diethylaminophenyl)spiro[2H-indole-2,3'-[3H]naphth[2,1,b]oxazine] (Reversacol Sea Green)
 - 1,3-dihydro-3,3-dimethyl-1-isobutyl-6'-(4-diethylaminophenyl)spiro[2H-

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indole-2,3'-[3H]naphth[2,1-b][1,4]oxazine] (Reversacol Aqua Green)

- 1,3-dihydro-3,3-dimethyl-1 isobutyl-6'-indolino spiro[2H-indole-2,3'-[3H] naphth[2,1-b][1,4]oxazine] (Reversacol Flat Purple)
- 1,3-dihydro-3,3-dimethyl-1-(2-phenylpropyl)-6'-indolino spiro[2H-indole-2,3'-[3H]naphth[2,1-b][1,4]oxazine] (Reversacol Storm Purple)
- 1,3-dihydro-3,3-dimethyl-1 isobutyl-6'-piperidino spiro[2H-indole-2,3'-[3H] naphth[2,1-b][1,4]oxazine] (Reversacol Plum Red)
- 1,3-dihydro-5-chloro-3,3-dimethyl-1 isobutyl-6'-piperidino spiro[2H-indole-2,3'-[3H]naphth[2,1-b][1,4]oxazine] (Reversacol Claret)

In a preferred aspect, one or more of the layers of the laminate optical layer may be subjected to a surface treatment to improve bondability and/or compatibility. The surface treatment may be selected from one or more of the group consisting of plasma discharge, corona discharge, glow discharge, ionising radiation, UV radiation, flame treatment and laser, preferably excimer laser treatment.

Preferably the inner layer is subjected to a plasma or corona discharge treatment.

In a further preferred aspect the optical laminate article may include a plurality of inner layers, each providing one or more desirable optical properties to the article. A combination of a photochromic inner layer and a polarising inner layer is particularly preferred.

The laminate optical article may further include standard additional coatings to the front or back surface.

The optical, preferably front lens, surface may include an anti-reflective (AR) coating, for example of the type described in United States Patent 5,704,692 to applicants, the entire disclosure of which is incorporated herein by reference. This is particularly suitable where the inner layer is a polarising film or foil. In conventional processing, when lens are AR coated they go through a stringent thermal regime, and this can damage the polarising foil. As the AR coating may be applied to a front and/or back lens wafer prior to assembly of the laminate article, thermal damage to the polarising film is simply avoided.

The optical, preferably front lens surface may include an abrasion

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resistant coating. e.g. of the type described in United States Patent 4,954,591 to applicants, the entire disclosure of which is incorporated herein by reference.

In a further preferred aspect of the present invention there is provided a series of laminate optical articles each member of the series including

a back lens wafer formed from a optical polymeric material;

an outer layer forming a protective coating; and

an inner layer positioned between said back lens wafer and outer protective layer, the inner layer providing a desirable optical property and shaped to have a curvature substantially corresponding to the interface surface of the back lens wafer.

Desirably successive members of the series may have suitable sphere and cylinder power to enable a member of the series to be used in combination with any design and power of front lens wafer to produce a finished lens within the normal tolerances for such lenses.

It is simply necessary to remove the protective coating just prior to attachment to the front lens wafer to provide a pristine and stable surface for lamination.

Alternatively, in a further preferred aspect, there is provided a laminate optical article including

a front lens wafer formed from a optical polymeric material;

an outer layer forming a protective coating; and

an inner layer positioned between said back lens wafer and outer protective layer, the inner layer providing a desirable optical property and shaped to have a curvature substantially corresponding to the interface surface of the front lens wafer.

The laminate optical article may further include a back lens wafer selected from a series of back lens wafers as described above.

Such back wafer series are described in United States Patent 5,187,505, to applicants, the entire disclosure of which is incorporated herein by reference.

The front and back lens wafer may further include one or more additions conventionally used in casting compositions such as inhibitors, dyes including electrochromic and photochromic dyes, e.g. as described above, polarising

agents. UV stabilisers and materials capable of modifying refractive index. Such additives may include:

UV Absorbers including

- Ciba Geigy Tinuvin P 2(2'-hydroxy-5'methyl phenyl) benzotriazole
- Cyanamid Cyasorb UV 531 -2-hydroxy-4-n-octoxybenzo- phenone 5
 - Cyanamid Cyasorb UV5411-2(2-hydroxy-5-t-octylphenyl)- benzotriazole
 - Cyanamid UV 2098 2 hydroxy-4-(2-acryloyloxyethoxy) benzophenone
 - National Starch and Chemicals Permasorb MA 2 hydroxy-4-(2 hydroxy-3- methacryloxy)propoxy benzophenone
- Cyanamid UV24 2,2'-dihydroxy-4-methoxybenzophenone 10
 - BASF UVINUL 400 2,4 dihydroxy-benzophenone
 - BASF UVINUL D-49 2,2'-dihydroxy-4,4' dimethoxy- benzophenone
 - BASF UVINUL D-50 2,2', 4,4' tetrahydroxy benzophenone
 - BASF UVINUL D-35-ethyl-2-cyano-3,3-diphenyl acrylate
- BASF UVINUL N-539-2-ethexyl-2-cyano-3,3-diphenyl acrylate 15
 - Ciba Geigy Tinuvin 213

Hindered amine light stabilisers (HALS), including

- Ciba Geigy Tinuvin 765/292 bis (1,2,2,6,6-pentamethyl-4-piperidyl) sebacate
- Ciba Geigy 770 bis (2,2,6,6-tetramethyl-4-piperidinyl) sebacate 20

Antioxidants including

- Ciba Geigy Irganox 245 triethylene glycol-bis-3- (3-tertbutyl-4-hydroxy-5methyl phenyl)propionate
- Irganox 1010 -2,2-bis[[3-[3,4-bis(1,1-dimethylethyl)-4-hydroxyphenyl]-1oxopropoxy]methyl]-1,3-propanediyl 3,5-bis(1,1-dimethyl ethyl)-4-hydroxy 25 benzene propanoate
 - 1076 octadecyl 3-(3',5'-di-tert-butyl(-4'- hydroxyphenyl) propionate

Anticolouring agents including

9. 10 dihydro-9-oxa-10-phosphaphenanthrene-1-oxide 30 Other monomeric additives may be present in, e.g. amounts up to 10% by

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weight as viscosity modifiers, and include monomers such as methacrylic acid, vinyl silanes, and other functional monomers. Other monomeric additives may be included to improve processing and/or material properties, these include:

- methacrylic acid, maleic anhydride, acrylic acid
- adhesion promoters/modifiers such as Sartomer 9008, Sartomer 9013,
 Sartomer 9015 etc.
 - dye-enhancing, pH-adjusting monomers like Alcolac SIPOMER 2MIM
 - a charge-reducing cationic monomer to render the material more antistatic, example Sipomer Q5-80 or Q9-75

The laminate optical article according to the present invention may be formed utilising any suitable techniques. Desirably, the laminate optical articles may be formed utilising a light curable adhesive, preferably a visible light or ultraviolet (UV) curable adhesive.

Accordingly, in a further preferred aspect there is provided a method for preparing a laminate optical article including

providing

a first outer layer formed from an optical polymeric material;

an inner layer formed from a polymeric film and capable of providing a desirable optical property to the final product; and

a light curable adhesive;

contacting at least a portion of a surface of the first outer layer with the light curable adhesive;

contacting a surface of the inner layer with the adhesive bearing surface of the first outer layer; and

curing the laminate so formed.

The light curable adhesive may be delivered to a surface of the first outer layer utilising any suitable technique. A syringe technique may be used.

The light curable adhesive may be of any suitable type. The light curable adhesive may be a visible light curable adhesive. The visible light curable adhesive may be selected from the group consisting of Dymax 469 and 492, Electro-Lite Corporation 4M12, Loctite 3321, Master Bond UV15-7 and 15X-2, and Lite-Fast Adhesive A-109 from Micro-Lite Technology.

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The adhesive may contact the surface generally centrally thereof, to provide a generally even spread of adhesive when contact with the inner layer is made. The adhesive coating step may continue for a period sufficient to permit the adhesive to spread over a substantial proportion of the surface. A period of 60 to 180 seconds is preferred.

Where the first outer layer is a front lens wafer, the contacting step may include orienting the inner layer relative to any characteristic feature or function of the front lens wafer. For example, the axis of polarisation should be orthogonal to the ledge on a flat top bifocal front.

In a preferred aspect, the curing step may include

subjecting the laminate to an initial cure whilst maintaining the components in contact under pressure for a time sufficient to achieve acceptable tack; and

subjecting the partially cured laminate to a final cure through the front and back surface thereof.

Preferably the initial and/or final cure is a visible light or UV cure.

In a further preferred aspect, the method may further include

providing a second outer layer formed from an optical polymeric material;

coating at least a portion of a surface of the second outer layer with the light curable adhesive;

contacting a surface of the inner layer with the adhesive bearing second outer layer; and

curing the laminate so formed.

Where the second outer layer is a back lens wafer, the contacting step may include orienting the cylinder on the back lens wafer according to the optical prescription requested.

A surface of one or more of the layers may be subjected to a surface treatment. Accordingly, in a preferred aspect of the present invention, the method may further include in a preliminary step a surface or part thereof of one or more of the layers is subjected to a surface treatment to improve bondability and/or compatibility.

The surfaces or parts of the surfaces of one or more of the layers making

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up the laminate may be modified to improve bondability and/or compatibility. The treatment may include physical and/or chemical modification of the surface of the polymer. These surface treatments may be selected from one or more of the group consisting of discharge treatment including plasma discharge, corona discharge, glow discharge, ionising radiation, UV radiation, flame treatment and/or excimer laser treatment. This treatment may be carried out in the presence of a suitable atmosphere, for example, oxygen, nitrogen, carbon dioxide, helium, argon and/or mixtures of these gases.

The treatment conditions and the time of treatment may be selected to suit the particular polymer type and other factors. The properties of the polymer surface may be altered by chemical means such as surface chemical grafting or by some other chemical treatment. A plasma discharge or corona discharge treatment is preferred. A low pressure air plasma treatment is particularly preferred.

The curing step may be a one step or two step process. The curing may occur after the inner layer is contacted with both the first outer layer and second outer layer. Alternatively the inner layer may be contacted with one outer layer and cured, after which the other outer layer is placed in contact with the inner layer and cured.

Accordingly in a still further preferred aspect, there is provided a method for preparing a laminate optical article including

providing

a first outer layer formed from an optical polymeric material; a second outer layer formed from an optical polymeric material; an inner layer with the adhesive bearing second outer layer; and a light curable adhesive;

coating at least a portion of a surface of the inner layer and one of the first or second outer layers with light curable adhesive;

contacting the adhesive bearing surfaces;

coating at least a portion of the other surface of the inner layer and the other of the first or second outer layers;

contacting the other adhesive bearing surfaces to form a laminate;

subjecting the laminate to an initial cure whilst maintaining the components in contact under pressure for a time sufficient to achieve acceptable tasks; and

subjecting the partially cured laminate to a final cure through the front and back surface thereof.

Desirably, the laminate may be reversed to complete the cure or the UV light, preferably visible or UV light may be directed from both sides of the laminate.

The present invention will now be more fully described with reference to the accompanying examples. It should be understood, however, that the description following is illustrative only and should not be taken in any way as a restriction on the generality of the invention described above.

EXAMPLES

Following is the description of the front and back lens wafer and inner layer components used in the following examples.

Front Polymeric Lens Wafer:

- 6.00 D interface curve
- Diallyl glycol carbonate material
- Hardcoat only

20 Back Polymeric Lens Wafer:

- 6.00 D interface curve
- Diallyl glycol carbonate material
- No coat

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Inner Polarising Film Layer:

25 • Preformed to 6.00 Base Curve

The lenses were subjected to the following tests:

Test Sample Description

All durability tests were performed on edged and surfaced plano samples, so as to ensure the most vulnerable test sample configuration.

The laminated 6.00 D base semi-finished polarising lenses were edged and surfaced as follows:

Lenses were diameter reduced (edged) from initial diameter 74 mm to final

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diameter 68 mm

Lenses were surfaced to 6.00 D base planos with centre thickness of 2.5
 mm.

Test Method Description

A sequence of steps was followed to assess the durability of laminated optical articles, by visual examination of any signs of delamination. Lenses were assessed after:

- Lamination completed
- Diameter reduction by edging
- 10 Thickness reduction by surfacing
 - 3 Hour Boil Test
 - 72 hours Atlas Weatherometer Humidity test

3 Hour Boil Test

Lenses were immersed in boiling water and examined visually for delaminations at 15 minute, 30 minute, 1 hours, 2 hours and 3 hours intervals. Additional % transmission and L*, a*, B* were measured at the intervals to illustrate overall durability of the polarising films.

Atlas Weatherometer Humidity Test

Lenses are stored in Atlas Weatherometer chamber for 72 hours, with inspection for delaminations every 24 hours. The conditions inside the Weatherometer are 65°C with 99% relative humidity. There is a two hours cycle time for increasing and decreasing temperature between 60-70°C.

Again, % transmission and colour coordinates were regularly checked as an indication of the polarising films' durability.

EXAMPLE 1

A UV curable adhesive was applied to a front polymeric lens wafer. A polyvinyl alcohol-type polarising film was then placed in contact with the wafer. The film is shaped to the curve of the laminate interface, typically 6D. The film was moved so that its edges align with the laminate element and properly oriented relative to any characteristic feature or function of the front lens wafer. For example, the axis of polarisation should be orthogonal to the ledge on a flat top bifocal front. Once the foil was correctly oriented with respect to the front

wafer, the cylinder on the back wafer was oriented according to the optical prescription required. Next, UV curable adhesive was placed on a back wafer placing it over the wafer element on which the polarising film has been placed. The axis was aligned and the adhesive UV cured as a standard lamination process. Once complete, the element may be reversed and given a supplementary UV treatment through the side that was shaded by the polarising film during the normal lamination operation. The lens was then edged and fitted. A lens of high quality and structural integrity was produced. No signs of delamination were observed.

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EXAMPLE 2

Lamination of Semi-Finished Polarising Lenses

A similar process was undertaken to that described in Example 1 above, with the following modifications.

Lamination Parts

The lamination process for semi-finished lenses consists of combining a front lens wafer with an inner polarising film layer and a semi-finished back component with adhesive in each of four interfaces.

EXAMPLE 3

The procedure of Example 1 was repeated to the point where the polarising film has been adhered to the front lens wafer. (Note: Some gentle pressure on the polarising film may be needed to get good conformity between it and the interface curve of the front laminate element). The front lens wafer bearing the polarising film or foil may then be packaged and shipped to be used as a standard laminate element. A finished polarising lens may then be produced by applying a back lens element at a later time.

EXAMPLE 4

The front lens wafer to which the polarising foil has been glued, as in Example 3, was subsequently coated with a peelable interface coating to protect the polarising film from physical damage. When peeled, just prior to lamination, a pristine surface is revealed, even if the external surface of the polarising film had been dirty at the time the interface coating was applied.

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EXAMPLE 5

Surface Treatment

The process of Example 1 was repeated except that the polarising film was subjected to a plasma treatment. The plasma treatment of polarising films was carried out in a Plasmod plasma system, manufactured by March Instruments (USA)

Following is a description of conditions used in plasma treatment.

Gas type :

Air

Vacuum

1 Torr

Power

30 - 50 W

Duration

10 - 60 sec

Low Pressure Plasma Treatment of Polarising Films - X-Ray Photo Electron

Spectroscopy (XPS) Analysis

The polarising film layers were formed of PVA based material impregnated with a chromophore. The films were exposed to low pressure air plasma for 60 seconds, and the surface of the films were analysed by XPS. General XPS survey scan indicates the types of elements present in the surface.

The following tables summarise the surface atomic concentrations obtained from general survey scans.

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<u>TABLE 1</u>
<u>Surface Atomic Concentrations</u>

Element	Before Plasma	After Plasma Conc (%)
	Conc (%)	
Carbon	70.5	62.5
Oxygen	29.5	36.4
Nitrogen	0	1.1

From the results it is obvious that oxygen concentration, as expected, has significantly increased after plasma treatment. Also, a small amount of nitrogen has been grafted onto the surface after plasma treatment.

Effect of Low Pressure Plasma Treatment on Durability of Laminated Lenses

The polarising films were exposed to low pressure air plasma for 60

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seconds. The treated films were laminated into a semi-finished product following standard laminating technique. The durability of the lens was compared to lenses, where films were laminated in untreated form. A visual examination for delaminations was performed after lamination, edging, surface, 3 Hour Boil test and 72 hours Humidity test.

Lenses laminated using plasma treated or untreated films during edging and surfacing could not be distinguished. There were no delamination defects determined in any of the lens samples studied.

However, during 3 Hour Boil test and 72 Hour Humidity test, differences between lenses became obvious.

Following table 2 summarises the results.

TABLE 2 3 Hour Boil Test (Polarising Film)

POLARISI	POLARISING FILM - UNTREAT	REATED				
Time	Initial	15 Min	30 Min	1 Hour	2 Hours	3 Hours
Delam.	No delam.	Delam. up to 5mm	Delam. increasing	Delam. not	Film further	Film further
		from edge	up to 10mm from	increas. Film	dissolving	dissolving
			edge	dissolving		
1%	17	16	16	17	18	20
<u>.</u>	48	47	47	49	20	52
**	-2.7	-2.7	-2.5	-2.3	-1.7	-0.40
,q	6.0+	+0.9	80.0-	-0.05	+0.50	-1.20
POLARISI	POLARISING FILM - PLASMA	SMA TREATED				
Time	Initial	15 Min	30 Min	1 Hour	2 Hours	3 Hours
Delam.	No Delam.	No Delam.	No Delam.	No Delam.	No Delam -	No Delam - Film
					Film slightly	retracted up to
					retracting	1mm
1%	16	16	17	17	18	22
*-	48	48	48	49	90	54
***	-2.6	-2.6	-2.8	-2.6	-1.30	-0.2
*q	+0.8	+0.5	+0.02	+0.2	-0.3	-2.2

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From results in table 2, it is obvious that lenses which were laminated using untreated polarising film started to delaminate after 15 minutes in boiling water. Most of the delaminations occurred within the first hour of boiling, which was then followed by total destruction of the polarising film within the delaminated areas.

Lenses which were laminated using plasma treated film did not delaminate during the entire 3 Hours Boiling test duration. Only slight film retraction is obvious after 2 hours of boiling.

The durability of lenses was studied in 72 Hours Humidity test. Lenses were checked visually for delaminations at 24 hour intervals. Table 3 summarises the results.

TABLE 3
Atlas Weatherometer Humidity Test

Film Type	Plasma Treatment	Initial	24 Hours	48 Hours	72 Hours
Polarising film	No	No delam	Delam up to 3mm around edges	Delam increasing up to 5mm	Film dissolving in delam area
Polarising film	Yes	No delam	No delam	- No delam	No delam

Again, the effect of plasma treatment is clearly obvious. Lenses laminated with treated film did not exhibit any delaminations, whilst lenses which were laminated using untreated film delaminated within 24 hours.

EXAMPLE 6

Comparative Polarising Lenses Durability Assessment

Laminated polarising lenses according to the present invention were compared to two prior art products, Talex (Japan) and Younger Optics (USA). The performance of the lenses was assessed using 72 hours Humidity test.

Visual assessment of delaminations were performed at 24 hour intervals, and colour coordinates were measured to examine overall durability of polarising films.

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The following table summarises the overall rating given to the durability performance of compared lenses.

Lens Type	Rating
Laminated Optical lens	1
Talex Grey	2
Talex Brown	2
Younger Optics	10

1 = Best, 10 = worst

Talex product and laminated optical lenses performed well in 72 hour Humidity test. Lenses manufactured by Talex did not show any signs of delaminations, however the polarising film retracted up to 2 mm from the edge of the lenses, after 72 hours exposure. The laminated lenses according to the present invention did not show any signs of delaminations and no film retractions from the edge. Lenses manufactured by Younger Optics started delaminating after 24 hours exposure. At the end of 72 hours exposure, the lenses were almost fully delaminated with severe substrate cracking in the front portion of the lenses.

The following table 5 shows the initial and final colour coordinates of tested lenses, in an attempt to illustrate the stability of the polarising films.

TABLE 5

Effect of 72 Hour Humidity Test on Colour Stability of the Film

Lens Type		Init	ial	,	Final			
	%T	L*	a*	b*	%T	L*	a*	b*
Laminated Optical	17	49	-2	-2	17	49	-1	-2
Talex Grey	10	37	+1	-3	11	39	+5	-1
Talex Brown	29	61	+3	+19	33	64	+5	+20
Younger Optics	39	68	-4	+7	67	86	-2	+1

The laminated optical lens according to the present invention exhibits the best stability, followed by films used in Talex product. However, film used in Younger Optics Products displayed a very poor temperature stability, graining up

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to 30% in transmission and completely losing colour.

EXAMPLE 7

a) <u>UV Curable Adhesive (Comparative)</u>

The process of Example 5 was repeated except that the front lens wafer was formed either from an inherently UV absorbing polymeric material or from a polymeric material modified to exhibit UV cutoff at a preselected wavelength (e.g. 320 nm to 380 nm).

The results are provided in Table 6 below.

TABLE 6
Effect of UV absorbing substrates on cure of Dymax 492

Cure Time			% Convers	sion	
(min)	UV320	UV350	UV370	UV380	Polarising film
0.2	10	2	0	. 0	0
0.4	38	12	0.2	0	0
0.6	59	26	0.4	0	0
0.8	74	39	0.7	0	0
1.0	83	49	1	0	0
2.0	92	79	7	0.2	3
5.0	94	90	35	3	35
10.0	97	92	62	12	69

90% conversion has to be achieved - otherwise cure inadequate

D492 - conventional UV cured adhesive

Conclusion

Dymax 492 cannot cure UV absorbing substrates

15 b) <u>Visible light curable adhesive</u>

The process of Example 7a was repeated except that a range of visible light curable adhesives were used.

The results are provided in Table 7 below.

TABLE 7

CR496 - Effect of UV absorbing substrates

				····	r		23		
Photochromic	Polarising	4	16	33	42	51	80	87	06
UV380 /	Polarising	င	13	26	38	46	71	85	88
Polycarbonate		5	22	39	51	59	62	87	. 06
Photochromic		5	24	41	53	62	82	68	91
Polarising		3	13	26	38	46	7.1	85	89
UV 380		9	29	47	09	70	84	88	06
UV 320		1	37	55	89	76	98	06	94
Cure Time	(min)	0.2	0.4	9.0	0.8	1.0	2.0	5.0	10.0

CR-496 - Proprietary SOLA visible adhesive

X279381 - commercial visible light adhesive

UV 380/polarising - means two layers

Photochromic/polarising - means two layers

Polycarbonate is inherently UV absorbing polymer

90% conversion achieved in all cases - cure adequate

TABLE 8

X-270-38-1 Effect of UV absorbing substrates

						24				
	Photochromic /	Polarising	5	22	38	48	59	8/	85	06
	UV 380 /	Polarising	~	9	14	23	32	57	62	87
ersion	Photochromic		7	28	46	57	65	83	89	91
% Conversion	Polarising Film		-	9	15	26	35	61	83	06
	UV380		8	35	54	99	75	86	06	94
	UV 320		8	34	51	64	73	88	92	97
Cure Time	(min)		0.2	0.4	0.6	0.8	1.0	2.0	5.0	10.0

Dymax X-279-38-1 - Commercially available visible adhesive Approximately 90% conversion achieved in all cases - cure adequate

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EXAMPLE 8

Laminate lens element bearing protective coating

Laminate lens elements comprising a front polymeric lens wafer and an inner polarising film layer were coated with a protective coating comprising a vinyl chloride/vinyl acetate copolymer dissolved in acetone and subsequently dried.

After three weeks there was no visible effect of the polarising film layer on the protective coating. There was no discolouration of the films and no evidence of dye transfer from the polarising film into the protective coating.

Effect of exposure of laminated semi-finished lenses to acetone

The laminated lens elements comprising a front polymeric lens wafer and an inner polarising film layer were soaked in acetone for 48 hours.

As acetone is a major component of a typical protective interface coating, the soaking process is an extreme test of the ability of the laminated lens element to withstand the protective coating process. For example, in a typical process the interface coating step continues for less than approximately one minute prior to drying.

After 48 hours, there was no obvious loss of colour in the polarising film and no obvious loss of polarisation. Further the acetone had not picked up any colour from the films.

Slight penetration of acetone along the glue layer was apparent but no delamination was apparent.

EXAMPLE 9

Lamination of Photochromic Lenses

A similar process was undertaken to that described in Example 1 above, with the following modifications.

The lamination process consists of laminating a front lens wafer with an inner photochromic layer and a back lens wafer with adhesive in each of four interfaces.

The inner photochromic layer comprises a cast flexible film, incorporating photochromic dyes. This film was shaped to match the interface curves on the front and back lens wafers.

The photochromic film is a polyoxyalkylene glycol dimethacrylate with

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urethane methacrylate including a mixture of three photochromic dyes. The photochromic dyes are selected from any of those described above. A typical mixture comprises a Reversacol Corn Yellow, Reversacol Berry Red and Reversacol Sea Green.

The laboratory lamination was performed using a standard lamination techniques. CR496 was used as the visible light curable adhesive.

The final cure of the adhesive was achieved by exposing the front and back surface of the laminated lens for four minutes (back surface).

A lens of high quality and structural integrity, exhibiting photochromic effects, was produced.

EXAMPLE 10

Lamination of Photochromic/Polarising Lenses

EXAMPLE 10A

A similar process was undertaken to that described in Example 1 above, with the following modifications.

The lamination process comprised laminating a front lens wafer with an inner shaped polarising film layer in the manner described in Example 1 above. The inner shaped polarising film layer was formed from a PVA material impregnated from a chromophore.

An inner photochromic layer as described in Example 9 above was laminated to the polarising film coated front lens wafer and a back lens wafer with adhesive in each of the four interfaces.

The laboratory lamination was performed using a standard lamination techniques. CR496 was used as the visible light curable adhesive.

The final cure of the adhesive was achieved by exposing the front and back surface of the laminated lens for four minutes (back surface).

EXAMPLE 10B

The process of Example 10A was repeated but the order of shaped polarising film layer and inner photochromic layer reversed.

In both examples, a lens of high optical quality and structural integrity, exhibiting a combination of photochromic and polarisation effects, was produced.

Finally, it is to be understood that various other modifications and/or

alterations may be made without departing from the spirit of the present invention as outlined herein.

Claims

- A laminate optical article including a first outer layer;
 a second outer layer; and
- an inner layer positioned between said first and second outer layers, the inner layer providing a desirable optical property to the laminate optical article.
 - 2... A laminate optical article according to Claim 1, wherein the first outer layer forms a front or back wafer of an optical lens and includes an optical surface and an interface or mating surface.
- 10 3. A laminate optical article according to Claim 2, wherein the first outer layer is formed from a glass or optical polymeric material.
 - 4. A laminate optical article according to Claim 3, wherein the first outer layer is formed from a polycarbonate material.
- 5. A laminate optical article according to Claim 1, wherein the second outer layer forms a protective coating for the inner layer.
 - 6. A laminate optical article according to Claim 5, wherein the protective coating includes one or more of the group consisting of an abrasion resistant component, a water resistant component and an antistatic component.
- 7. A laminate optical article according to Claim 2, wherein the second outer layer forms a complementary back or front wafer of a laminate optical lens.
 - 8. A laminate optical article according to Claim 7, wherein the second outer layer is formed from a glass or optical polymeric material.
 - 9. A laminate optical article according to Claim 8, wherein the second outer layer is formed from a polycarbonate material.
- 25 10. A laminate optical article according to Claim 1, wherein the inner layer includes a polymeric film or foil having desirable optical properties.
 - 11. A laminate optical article according to Claim 10, wherein the inner layer has a shape complementary to a surface of the first and/or second outer layers.
- 12. A laminate optical article according to Claim 11, wherein the inner layer
 30 has a curvature which substantially corresponds to the curvature of the inner
 surface of the first and/or second outer layer.
 - 13. A laminate optical article according to Claim 10, wherein the polymeric

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film or foil includes an active light polarising film.

- 14. A laminate optical article according to Claim 10, wherein the polymeric film or foil has incorporated therein an optically active component selected from the group consisting of one or more of the group consisting of light polarising materials, ultra-violet inhibitors, photochromic materials, electrochromic materials and other pigments or dyes.
- 15. A laminate optical article according to Claim 14, wherein the inner layer includes a photochromic dye(s) selected from one or more of the group consisting of anthraquinones, phthalocyanines, spiro-oxazines, chromenes, pyrans including spiro-pyrans and fulgides.
- 16. A laminate optical article according to Claim 1, including a plurality of inner layers each providing one or more desirable optical properties to the laminate optical article.
- 17. A laminate optical article according to Claim 16, including an inner polarising film layer and an inner photochromic layer.
 - 18. A laminate optical article according to Claim 2, wherein an optical surface thereof includes an anti-reflective (AR) coating and/or abrasion resistant coating thereon.
- 19. A laminate optical article according to Claim 1, wherein a surface or part thereof of one or more of the layers is subjected to a surface treatment to improve bondability and/or compatibility.
 - 20. A laminate optical article according to Claim 19, wherein the surface treatment is selected from one or more of the group consisting of plasma discharge, corona discharge, glow discharge, ionising radiation, UV radiation, flame treatment and laser treatment.
 - 21. A laminate optical article according to Claim 20, wherein the inner layer is subjected to a plasma or corona discharge treatment.
 - 22. A laminate optical article including a first front lens wafer formed from a optical polymeric material;
- a second complementary back lens wafer formed from a optical polymeric material; and

an inner layer positioned between said front and back lens wafer and

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having a shape complementary to a surface of the front and/or back wafer, the inner layer providing a desirable optical property to the laminate optical article.

- 23. A series of laminate optical articles, each member of the series including a back lens wafer formed from a optical polymeric material;
- 5 an outer layer forming a protective coating; and
 - an inner layer positioned between said back lens wafer and outer protective layer, the inner layer providing a desirable optical property and shaped to have a curvature substantially corresponding to the interface surface of the back lens wafer.
- 10 24. A laminate optical article series according to Claim 23, wherein the protective coating is formed from a vinyl chloride/vinyl acetate copolymer.
 - 25. A laminate optical article series according to Claim 23, wherein successive members of the series have suitable sphere and cylinder power to enable a member of the series to be used in combination with any design and power of front lens wafer to produce a finished lens within the normal tolerances for such lenses.
 - 26. A laminate optical article including: a front lens wafer formed from a optical polymeric material; an outer layer forming a protective coating; and
- an inner layer positioned between said back lens wafer and outer protective layer, the inner layer providing a desirable optical property and shaped to have a curvature substantially corresponding to the interface surface of the front lens wafer.
 - 27. A laminate optical article according to Claim 26, wherein the protective coating is formed from a vinyl chloride/vinyl acetate copolymer.
 - 28. A laminate optical article according to Claim 26, further include a back lens wafer selected from a series of back lens wafers according to Claim 22.
 - 29. A method for preparing a laminate optical article including: providing
 - a first outer layer formed from an optical polymeric material; an inner layer formed from a polymeric film and capable of providing a desirable optical property to the final product; and

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a light curable adhesive;

coating at least a portion of a surface of the first outer layer with the light curable adhesive;

contacting a surface of the inner layer with the adhesive bearing surface of the first outer layer; and curing the laminate so formed.

- 30. A method according to Claim 29, wherein the light curable adhesive is delivered to a surface of the first outer layer generally centrally thereof, utilising a syringe technique, to provide a generally even spread of adhesive when contact with the inner layer is made.
- A method according to Claim 30, wherein the adhesive coating step continues for a period sufficient to permit the adhesive to spread over a substantial proportion of the surface.
- 32. A method according to Claim 29, wherein the curing step includes:
- subjecting the laminate to an initial cure whilst maintaining the components in contact under pressure for a time sufficient to achieve acceptable tack; and

subjecting the partially cured laminate to a final cure through the front and back surface thereof.

- 20 33. A method according to Claim 32, wherein the initial and/or final cure is a visible light or UV cure.
- 34. A method according to Claim 29, further including providing a second outer layer formed from an optical polymeric material; coating at least a portion of a surface of the second outer layer with the light curable adhesive;

contacting a surface of the inner layer with the adhesive bearing second outer layer; and

curing the laminate so formed.

- 35. A method according to Claim 29, wherein in a preliminary step, a surface or part thereof of one or more of the layers is subjected to a surface treatment to improve bondability and/or compatibility.
 - 36. A method according to Claim 35, wherein the inner layer is subjected to a

plasma or corona discharge treatment.

37. A method for preparing a laminate optical article including:

providing a first outer layer formed from an optical polymeric material:

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a second outer layer formed from an optical polymeric material; an inner layer with the adhesive bearing second outer layer; and a light curable adhesive;

coating at least a portion of a surface of the inner layer and one of the first or second outer layers with light curable adhesive;

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contacting the adhesive bearing surfaces;

coating at least a portion of the other surface of the inner layer and the other of the first or second outer layers with light curable adhesive;

contacting the other adhesive bearing surfaces to form a laminate subjecting the laminate to an initial cure whilst maintaining the components in contact under pressure for a time sufficient to achieve acceptable tack; and

subjecting the partially cured laminate to a final cure through the front and back surface thereof.

38. A laminate optical article substantially as hereinbefore described with reference to any one of the examples.

INTERNATIONAL SEARCH REPORT

International Application No. PCT/AU 96/00805

Α.	CLASSIFICATION OF SUBJECT MATTER				
Int Cl ⁶ : G02	2B 1/00, 1/04, G02C 7/02, B29D 11/00				
	International Patent Classification (IPC) or to both	national classification and IPC			
В.	FIELDS SEARCHED				
Minimum docui IPC G02B-1/	mentation searched (classification system followed by c 700, 1/04, G02C 7/02, B29D 11/00	lassification symbols)			
Documentation AU: IPC as	searched other than minimum documentation to the extabove	tent that such documents are included in t	he fields searched		
	base consulted during the international search (name of Lamina and Lens	f data base and, where practicable, search	terms used)		
C.	DOCUMENTS CONSIDERED TO BE RELEVANT	[
Category*	Citation of document, with indication, where app	propriate, of the relevant passages	Relevant to claim No.		
х	AU, 78350/94 A (POLAROID CORPORATION) 3 April 1995				
x	AU, 77045/94 A (IMPERIAL CHEMICAL IND page 11, lines 29-34	USTRIES PLC) 1 May 1995	29-33		
X Y	AU, 67066/94 A (SOL A GROUP LTD) 8 Nove page 3, line 30 - page 4, line 27	mber 1994	1-12,14,16,18,22,23,26,28 25		
x	Further documents are listed in the continuation of Box C	X See patent family annex			
* Special categories of cited documents: "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier document but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published after the international filing date or understand the principle or theory underlying the invention document of particular relevance; the claimed invention can be considered novel or cannot be considered to involve an inventive step when the document of particular relevance: the claimed invention can be considered to involve an inventive step when the document of particular relevance: the claimed invention can be considered to involve an inventive step when the document of particular relevance: the claimed invention can be considered to involve an inventive step when the document of particular relevance invention can be considered to involve an inventive step when the document of particu					
	out later than the priority date claimed ual completion of the international search	Date of mailing of the international sear	ch report		
7 March 1997		1 4 MAR 1997			
Name and mail AUSTRALIAN PO BOX 200 WODEN ACT AUSTRALIA	ling address of the ISA/AU I INDUSTRIAL PROPERTY ORGANISATION 2606 Facsimile No.: (06) 285 3929	Authorized officer M. E. DIXON Telephone No. (06) 283 2194			

AU 96/00805INTERNATIONAL SEARCH REPORT

International Application No.
PCT/AU 96/00805

C (Continua	tion) DOCUMENTS CONSIDERED TO BE RELEVANT	_
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Y	AU, 28396/92 (647324) B (PILKINGTON VISIONCARE INC) 20 May 1993 page 4, lines 20-26, page 7, line 17 - page 9, line 22	25
x	AU, 54934/73 (478042) B (POLAROID CORPORATION) 31 October, 1974 page 7, line 27 - page 12, line 8, page 18, lines 5-18	1-3,5-8,10- 14,19,22,23,25 ,26,28
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x	GB, 2048776 A (CORNING GLASS WORKS) 17 December 1980 Abstract, Fig 4	1- 8,19,22,23,26, 28
P,X	WO, 96/27496 A (BMC INDUSTRIES, INC.) 12 September 1996 Whole specification	1-19,22-35,37
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